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Purple wilfordii insecticide and its preparing process

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Inventor: LIN SUI (CN)
Applicant: FUJIAN INST OF MEDICAL SCIENCE (CN)
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Abstract of CN1317248

An insecticide "Zileiteng" contains TW1, TW2 and TW3 in weight ratio of 1:(0.8-1.2):(0.8-1.2), which are extracted from the raw wilfordii through seeping, silica gel column chromatography and eluting. Its advantages include simple preparing process, high stability and no environmental pollution.

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Fujian Province Medical Research Institute

A MILLETTIA ATROPURPUREA¹ PESTICIDE AND PREPARATION METHOD THEREOF

[54] Title of Invention: A Millettia Atropurpurea Pesticide and Preparation Method
Thereof

[57] Abstract. This invention discloses a green pesticide whose main component includes extracts TW_1 , TW_2 and TW_3 of the raw medicinal herb *Tripterygium wilfordii* Hook f. at a weight ratio of $TW_1 : TW_2 : TW_3$ equal to 1:0.8~1.2:0.8~1.2. Alternatively, the principal components include TW_1 , TW_2 and TW_3 . The described preparation method involves taking the raw herb *Tripterygium wilfordii* Hook f., percolating, extracting it and eluting on a silica gel column for analysis and separation, thus obtaining the extract TW_1 . Alternatively, the raw herb *Tripterygium wilfordii* Hook f. is taken, percolated, extracted and eluted with different liquid eluants, thus collecting the components TW_2 and TW_3 . Therefore this is a pesticide with a straightforward technological process; it is a stable product and a pesticide harmless to the public.

CLAIMS

1. A green pesticide whose main component includes extracts TW_1 , TW_2 and TW_3 of the raw medicinal herb *Tripterygium wilfordii* Hook f. at a weight ratio of $TW_1 : TW_2 : TW_3$ equal to 1:0.8~1.2:0.8~1.2.
2. A green pesticide whose main component includes extracts TW_1 , TW_2 or TW_3 of the raw medicinal herb *Tripterygium wilfordii* Hook f.
3. The method of preparing the green pesticide described in Claim 1 or Claim 2 characterized in that raw medicinal herb *Tripterygium wilfordii* Hook f. is taken, it is percolated and extracted with added ethanol, obtaining ethanol extract, extracted with chloroform to obtain a chloroform extract and subjected to silica gel column chromatography, then eluted with mineral ether : ethyl acetate to obtain the extract TW_1 .
4. The method of preparing the green pesticide described in Claim 1 or Claim 2 characterized in that raw medicinal herb *Tripterygium wilfordii* Hook f. is taken, it is percolated and extracted with added ethanol, obtaining ethanol extract, extracted with ethyl ether to obtain an ethyl ether extract, subjected to silica gel column chromatography, then eluted with ethyl ether, to collect fractions containing TW_2 and TW_3 , which then are separately combined, again subjected to silica gel column chromatography, and eluted with chloroform : acetone to collect components TW_2 and TW_3 .

¹ This translation is an unsupported guess. The combination 紫雷藤 (zileiteng) gets only 3 hits on Google and the plant is referred in the text of the patent under a different name 雷公藤 (leigongteng) for which the translation is *Tripterygium wilfordii* Hook f. – Translator's note.

5. The method of preparing the green pesticide described in Claim 1 or Claim 2 characterized in that when the extract of the raw medicinal herb *Tripterygium wilfordii* Hook f. is taken to prepare the pesticide, the content ratio of the TW_1 , TW_2 and TW_3 extracts is 1:0.8~1.2:0.8~1.2, and the content of the three components TW_1 , TW_2 or TW_3 in the extract is 1/500.

Specifications

A Millettia Atropurpurea Pesticide and Preparation Method Thereof

This invention pertains to a plant pesticide and preparation method thereof.

Tripterygium wilfordii Hook f. (Celastraceae) is a *Tripterygium* woody climber plant. Its main medicinal parts are roots, leaves, flowers, and fruit. Its pharmacological effects that have so far been reported in medical circles include anti-rheumatic, anti-tumor, immunosuppressive, and contraceptive, etc. There have been no reports of it being individually used as a pesticide in agriculture.

The purpose of this invention is to offer a green pesticide against plant-destroying pests using a *Tripterygium wilfordii* Hook f. extract.

The goal of this invention is achieved in the following manner. The main component of said green pesticide includes extracts TW_1 , TW_2 and TW_3 of the raw medicinal herb *Tripterygium wilfordii* Hook f. at a weight ratio of $TW_1 : TW_2 : TW_3$ equal to 1:0.8~1.2:0.8~1.2. The method of preparation of this pesticide calls for combining the extracts of the raw medicinal herb *Tripterygium wilfordii* Hook f. TW_1 , TW_2 , and TW_3 at a weight ratio of 1:0.8~1.2:0.8~1.2 and then adding other components of *Tripterygium wilfordii* Hook f. herb to make a mixture. The three components TW_1 , TW_2 , and TW_3 account for 1/500 of the mixture. Alternatively the main component of said green pesticide includes the extracts of the raw medicinal herb *Tripterygium wilfordii* Hook f. TW_1 , TW_2 , or TW_3 . For the raw medicinal herb *Tripterygium wilfordii* Hook f. TW_1 extract, the raw medicinal herb *Tripterygium wilfordii* Hook f. is taken, it is percolated and extracted with added ethanol, obtaining ethanol extract, extracted with chloroform to obtain a chloroform extract and subjected to silica gel column chromatography, then eluted with mineral ether : ethyl acetate to obtain the extract TW_1 . It contains five-member unsaturated lactone ring, three-member epoxides, triple six-member rings, hydroxyl groups, and other groups; it is diluted by a factor of 800~1000 with water and its toxic rate with regard to the *Wulingcai* green worm and *Helicoverpa Armigera* is as high as 93.6%. For the raw medicinal herb *Tripterygium wilfordii* Hook f. TW_2 and TW_3 extracts, the raw medicinal herb *Tripterygium wilfordii* Hook f. is taken, it is percolated and extracted with added ethanol, obtaining ethanol extract, extracted with ethyl ether to obtain an ethyl ether extract, subjected to silica gel column chromatography, then eluted with ethyl ether, to collect fractions containing TW_2 and TW_3 , which then are separately combined, again subjected to silica gel column chromatography, and eluted with chloroform : acetone to collect components TW_2 and TW_3 . They mainly contain a six-member pyridine ring, sesquiterpene lactone, hydroxyl group, and other groups; TW_2 and TW_3 extracts are diluted with water by a factor of 800~1000. Of these components TW_2 has an anti-feeding effect both on *Wulingcai* green worm and *Helicoverpa Armigera*, with an anti-feeding rate of 86.7%; TW_3 has a narcotic effect both on *Wulingcai* green worm and *Helicoverpa Armigera* with a narcotic

rate of 89.5%. Due to the agrochemical toxicological combination, as shown by application and follow-up experiments, the three compounds have a unique mechanism of pesticidal effect. The larva, apart from slow movements and manifested lethargy, develop black spots on the surface of their bodies and ever more deformed bodies as well as a number of pathological changes; field experiments on their defensive effectiveness demonstrate a very strong protective effect. Experiments prove that they have a strong pesticidal activity against common pests attacking tea leaves, tobacco leaves, vegetables, cotton, and other cash crops and edible crops. Furthermore, the dose of the three compounds as pesticides is extremely low, and they are not harmful to humans (toxic dose for humans is >10 micrograms/kg, and the dose absorbed after the release of the pesticide is <1 microgram/kg.)

Since this invention uses natural plant *Tripterygium wilfordii* Hook f. as a pesticide, it cannot release new chemicals into nature or pollute the environment by pesticide residues, thus it falls under chemicals that pose no harm to public health; field experiments on its defensive efficiency also prove that it has a very strong protective effectiveness; its dose as a pesticide is extremely low, and it is not harmful to humans.

Below, we will further explain this invention in combination with Practical Examples:

In the Practical Examples below, the inner diameter of the silica gel column is 6 cm; it was manufactured at the Great Wall Instrument Plant at Zhengzhou. The reagents used are reagents for industrial use. The process of manufacturing the raw medicinal herb *Tripterygium wilfordii* Hook f. was: *Tripterygium wilfordii* Hook f. root skin was crushed.

Practical Example 1

10 kg of the raw medicinal herb *Tripterygium wilfordii* Hook f. was taken, and, upon adding an appropriate amount of industrial ethanol, it was percolated and extracted, thus obtaining an ethanol extract; this was extracted with an appropriate amount of chloroform, thus obtaining a chloroform extract, and, upon subjecting to silica gel chromatography, it was eluted with mineral ether : ethyl acetate (mol ratio 1:1), yielding the extract TW_1 . It contained It contains five-member unsaturated lactone ring, three-member epoxides, triple six-member rings, hydroxyl groups, and other groups, and as a pesticide, it was diluted with water by a factor of 800~1000; its toxic rate with regard to the Wulingcai green worm and *Helicoverpa Armigera* was as high as 93.6%.

Practical Example 2

10 kg of the raw medicinal herb *Tripterygium wilfordii* Hook f. was taken, and, upon adding an appropriate amount of industrial ethanol, it was percolated and extracted, thus obtaining an ethanol extract; this was extracted with an appropriate amount of ethyl ether, thus obtaining an ethyl ether extract, upon subjecting it to silica gel chromatography, it was eluted with an appropriate amount of ethyl ether, and fractions of TW_{II} and TW_{III} were collected, the separately combined and again subjected to silica gel chromatography, upon elution with an appropriate amount of chloroform : acetone (mol ratio 1:1), components TW_{II} and TW_{III} were collected. They mainly contained a six-member pyridine ring, sesquiterpene lactone, hydroxyl group, and other groups; when

used as pesticides, TW_{II} and TW_{III} extracts were diluted with water by a factor of 800~1000. Of these components, TW_{II} had an anti-feeding effect both on Wulingcai green worm and *Helicoverpa Armigera*, with an anti-feeding rate of 86.7%; TW_{III} had a narcotic effect both on Wulingcai green worm and *Helicoverpa Armigera* with a narcotic rate of 89.5%.

Practical Example 3

The *Tripterygium wilfordii* Hook f. extract contained extracts TW_I , TW_{II} , and TW_{III} at a ratio of 1 : 1 : 1; the content of the three components TW_I , TW_{II} , and TW_{III} in the extract was 1/500, and these three components were used as a quality control index. When used as a pesticide, they had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to them.

Practical Example 4

The *Tripterygium wilfordii* Hook f. extract contained extracts TW_I , TW_{II} , and TW_{III} at a ratio of 1 : 0.8 : 0.9; the content of the three components TW_I , TW_{II} , and TW_{III} in the extract was 1/500, and these three components were used as a quality control index. When used as a pesticide, they had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to them.

Practical Example 5

The *Tripterygium wilfordii* Hook f. extract contained extracts TW_I , TW_{II} , and TW_{III} at a ratio of 1.1 : 1.1 : 1.2; the content of the three components TW_I , TW_{II} , and TW_{III} in the extract was 1/500, and these three components were used as a quality control index. When used as a pesticide, they had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to them.

Practical Example 6

The *Tripterygium wilfordii* Hook f. extract contained extracts TW_I , TW_{II} , and TW_{III} at a ratio of 1 : 0.9 : 1.2; the content of the three components TW_I , TW_{II} , and TW_{III} in the extract was 1/500, and these three components were used as a quality control index. When used as a pesticide, they had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to them.

Practical Example 7

The *Tripterygium wilfordii* Hook f. extract contained extracts TW_I , TW_{II} , and TW_{III} at a ratio of 1 : 1.1 : 0.9; the content of the three components TW_I , TW_{II} , and TW_{III} in the extract was 1/500, and these three components were used as a quality control index. When used as a pesticide, they had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to them.

Practical Example 8

The *Tripterygium wilfordii* Hook f. extract contained extract TW_I ; the content of the component TW_I in the extract was 1/500, and this component was used as a quality control index. When used as a pesticide, it had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to it.

Practical Example 9

The *Tripterygium wilfordii* Hook f. extract contained extract TW_{II} ; the content of the component TW_{II} in the extract was 1/500, and this component was used as a quality control index. When used as a pesticide, it had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to it.

Practical Example 10

The *Tripterygium wilfordii* Hook f. extract contained extract TW_{III} ; the content of the component TW_{III} in the extract was 1/500, and this component was used as a quality control index. When used as a pesticide, it had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to it.